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14. ABSTRACT

The microwave absorption characteristics of gold nanorod particles in aqueous solution were explored in two sets of experiments. The first experiments employed heating as an indirect indicator of microwave absorption at 4 GHz in a waveguide setup. Several solutions using gold nanoparticles in concentrations up to 5 mM with two different particle aspect ratios up to 4-to-1 showed little heating attributable to the nanoparticles but some heating attributable to CTAB in the solution, which is necessary in synthesis to prevent particle aggregation. Swept open-ended coaxial probe measurements from 0.5 to 20 GHz and spotfrequency cavity measurements at 2.19 and 5 GHz likewise showed little absorption attributable to the gold nanoparticles. The results suggest that small aspect ratio gold nanoparticles at low concentrations are likely not highly effective microwave absorbers. Larger aspect ratio gold nanoparticles may still hold promise as microwave theranostic agents.

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Gold nanoparticles, gold nanorods, microwave properties, theranostic agent, breast cancer, microwave imaging.

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ABSTRACT:

The microwave absorption characteristics of gold nanorod particles in aqueous solution were explored in two sets of experiments. The first experiments employed heating as an indirect indicator of microwave absorption at 4 GHz in a waveguide setup. Several solutions using gold nanoparticles in concentrations up to 5 mM with two different particle aspect ratios up to 4-to-1 showed little heating attributable to the nanoparticles but some heating attributable to CTAB in the solution, which is necessary in synthesis to prevent particle aggregation. Swept open-ended coaxial probe measurements from 0.5 to 20 GHz and spot-frequency cavity measurements at 2.19 and 5 GHz likewise showed little absorption attributable to the gold nanoparticles. The results suggest that small aspect ratio gold nanoparticles at low concentrations are likely not highly effective microwave absorbers. Larger aspect ratio gold nanoparticles may still hold promise as microwave theranostic agents.

I) Summary of Experimental Data: Waveguide Heating

A) Initial experiments suggesting enhanced heating due to gold nanorods in aqueous solution with CTAB

Part 1: Description of Particles

Three kinds of samples are used in the experiment: DI water, nanorods with plasmon peak at 762 nm and nanorods with plasmon peak at 841 nm. Both of the nanorods will be referred to as 762 nanorods and 841 nanorods in the rest of the document.

Part 2: Experiment description

The microwave frequency is at 4 GHz, and the output power from the amplifier is adjusted to 7 watt. At the optimal position of the tuner, the VSWR value is around 1.1. The power level is monitored using a directional coupler and power meter during the heating cycle to ensure stability. Each experiment trial consists of a set of three heating and cooling cycles and three trials are performed for each sample (a total of nine heating/cooling cycles for each sample). A heating cycle lasts 3 minutes, and a cooling cycle lasts 7 minute. Each experiment trial lasts for 30 minute total (each sample is tested for 90 minutes).

Part 3: Verification

Three trials were tested for DI water to ensure there is repeatability. Subsequently, three trials were performed for both the 5mM concentration of the 841 nanorods and 5 mM concentration of the 762 nanorods for repeatability. Figure 1 provides the illustration for the heating and cooling cycles curves. Note that for DI water and the 841 nanorods, the heating and cooling curves do not overlap exactly between different trials. That is due to a slight fluctuation in the room temperature, as can be seen in the starting point of the heating cycle. The room temperature can fluctuate by one degree depending on the time of the day. However, conductivity is related to the temperature difference between the room temperature and the temperature the samples asymptotically approach to during the heating cycle. As long as the temperature difference can be shown to be consistent, the trials are shown to be repeatable. Table 1 shows the temperature difference is fairly consistent for all samples during three separate experiment trials. The data fitting used to retrieve the temperature information is described in the next part.

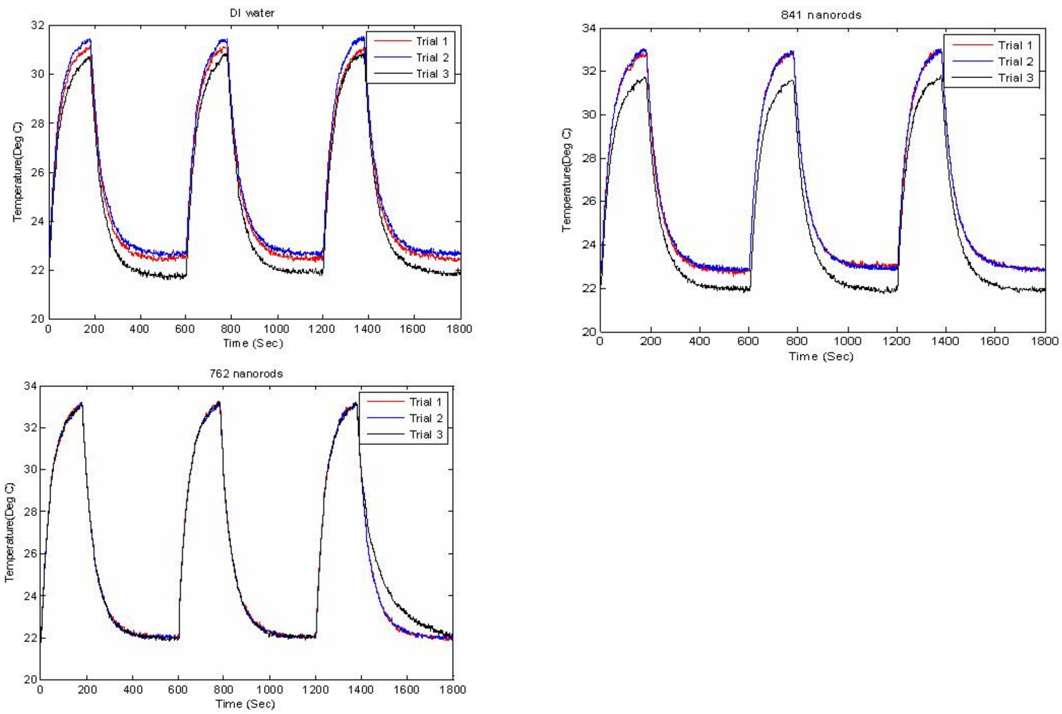


Figure 1 Heating and cooling cycles of DI water, 841 plasmon peak nanorods and 762 plasmon peak nanorods

Samples	**Trial 1**	**Trial 2**	**Trial 3**	**AVERAGE**
DI Water	8.665267	8.839767	8.9908	8.831944
762 nanorods 5mM	11.19513	11.1049	11.127	11.14234
841 nanorods 5mM	10.06773	10.1083	9.771533	9.982522

Table 1: Difference between room temperature and the temperature sample asymptotically approaches to during the heating cycle. Three trials are performed to ensure the repeatability of the experiment

Part 4: Results

DI water and several concentrations (5mM, 2.5 mM, and 1.25 mM) of the 762 nanorods and 841 nanorods are used for the heating experiment. The samples are diluted with DI water. Exponential functions are used to fit the temperature trace data, and they take the form.

$$\text{Heating phase: } T(t) = a_1 - b_1 \exp(-t/\tau_1) \qquad (1a)$$

$$\text{Cooling phase: } T(t) = a_2 + b_2 \exp(-t/\tau_2)$$

a_2 is equivalent to the room temperature, and a_1 is the temperature the samples asymptotically approach during the heating cycle. Conductivity is proportional to $\sigma \propto (a_1 - T_0)$, and $(a_1 - T_0)$ is the normalized conductivity. The normalized conductivity is listed in Table 2 for DI water, 762 nanorods and 841 nanorods at different concentrations. The general trend is that both samples of nanorods have a higher normalized conductivity than DI water and hence have a more efficient heating effect than DI water. Figure 2 and Figure 3 show the temperature trace data of different concentrations of both kinds of nanorods

Samples	Average Normalized conductivity
DI Water	8.831944
762 nanorods 5mM	11.14234
762 nanorods 2.5mM	10.62992
762 nanorods 1.25mM	10.1621
841 nanorods 5mM	9.982522
841 nanorods 2.5mM	9.534856
841 nanorods 1.25mM	9.513946

Table 2: Comparison of the normalized conductivity between DI water, 762 nanorods and 840 nanorods at 5mM, 2.5 mM and 1.25 mM

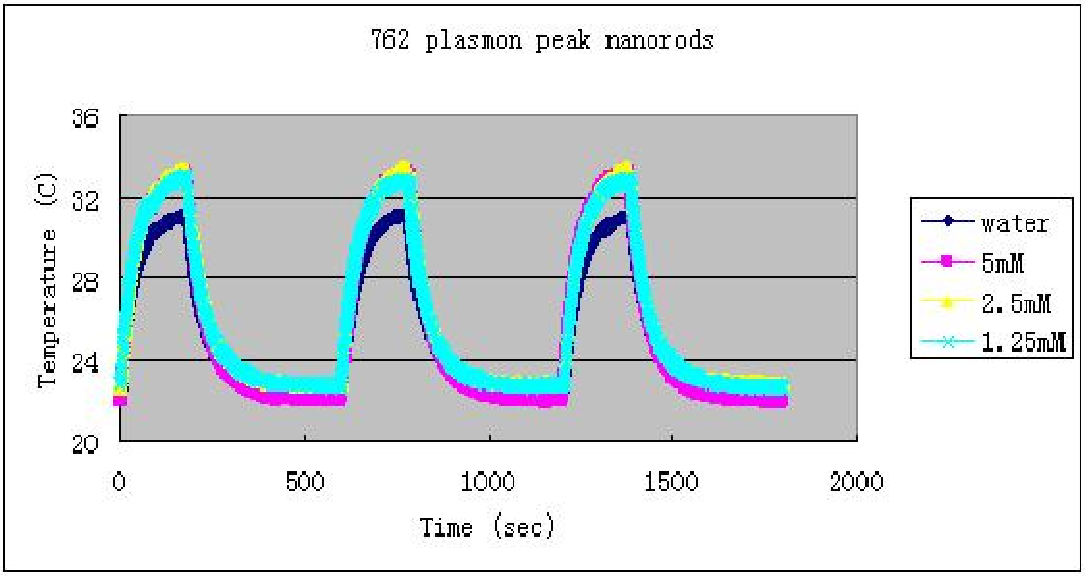


Figure 2: Temperature trace of different concentrations of 762 nanorods

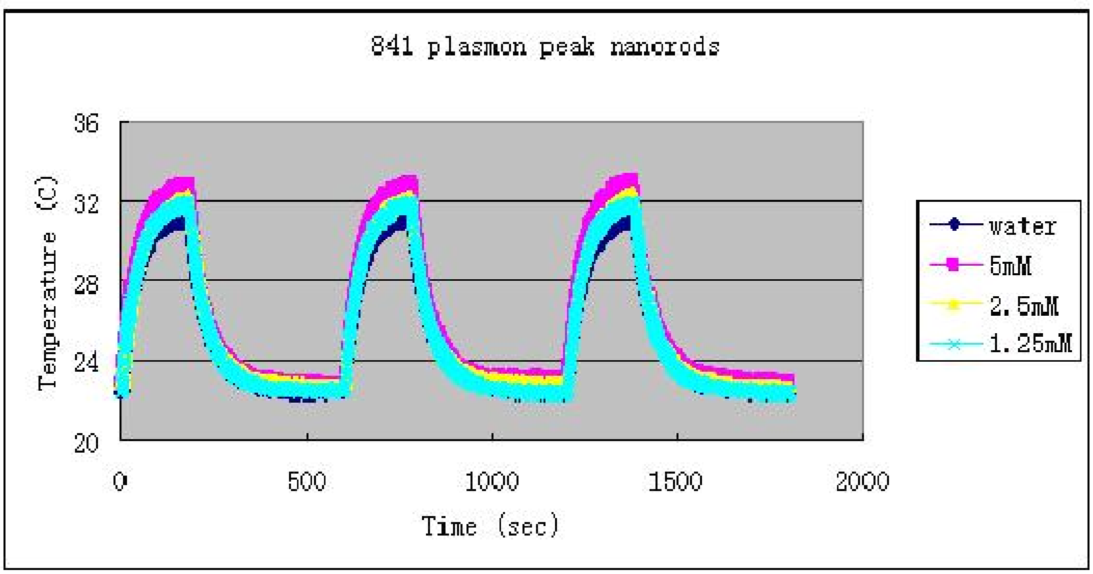


Figure 3: Temperature trace of different concentrations of 841 nanorods

A heating effect is observed in both of the nanoparticles samples (762 nanorods and 841 nanorods) compared to DI water, and the effect is related to the concentration of the nanoparticles. {Note that subsequent experiments, reported below, suggest that heating may be due to CTAB in solution not the gold nanoparticles}.

B) Summary of Data of Microwave Heating Experiment

DI water and several concentrations of nanorods with plasmon peak at 762 nm and 841 nm are used for the heating experiment. **The two kinds of nanorods will be referred to as 762 nanorods and 841 nanorods in the rest of the document.** The samples are diluted with DI water from the original 5 mM concentrations of particles that Kvar Black (in the Messersmith lab) made. Exponential functions are used to fit the temperature trace data, and they take the form.

$$\text{Heating phase: } T(t) = a_1 - b_1 \exp(-t/\tau_1) \qquad (1)$$
$$\text{Cooling phase: } T(t) = a_2 + b_2 \exp(-t/\tau_2)$$

a_2 is equivalent to the room temperature, and a_1 is the temperature the samples asymptotically approach during the heating cycle. Conductivity is proportional to $\sigma \propto (a_1 - T_0)$, and $(a_1 - T_0)$ is the normalized conductivity. There are two sets of data listed in this report, one set of data taken before UW staff came to NU and one set taken when UW staff were at NU.

Each experiment trial consists of a set of three heating and cooling cycles and three trials are performed for each sample. A heating cycle lasts 3 minutes, and a cooling cycle lasts 7 minute. Each experiment trial lasts for 30 minute total.

Part I: Data taken before UW apparatus was brought to NU.

This set of data includes DI water and three concentrations (5mM, 2.5 mM, and 1.25 mM) of 762 nanorods and 841 nanorods. This is the same set of data sent before the UW staff visited NU. In general, 3 trials were performed for each sample. Table 1a is a summary of the data comparing the normalized conductivity of DI water and the two kinds of nanorods. The table shows the average normalized conductivity of all the trials taken for each kind of samples, standard deviation between the trials and the 95% confidence interval. Figure 1 shows a plot of the average normalized conductivity for all the trials vs. concentration for each sample, and the 95 % confidence interval is also included. The general trend is that both samples of nanorods have a higher normalized conductivity than DI water and hence have a more efficient heating effect than DI water.

Two sample t-test has been performed to compare the mean of the normalized conductivity from water and from the 5mM concentration of each kind of nanorods. Three trials were performed for each sample. The mean and standard deviation of the trials were determined for them, and the two-sample t-test is used to determine whether the trial means indeed differ.

Table 1b shows the two samples t-test performed for the normalized conductivity comparing DI water with 5 mM concentration of 762 nanorods, and Table 1c shows the t-test comparing DI water with 5 mM concentration of 841 nanorods. For the t-test comparing DI water with the 762 nanorods, the two-tailed p-value is less than 0.0001. Generally, if the p-value is less than 0.05, the two sample means difference can be concluded to be statistically significant. It can be concluded that the normalized conductivity mean is different between DI water and 762 nanorods, and that the 762 nanorods has a higher normalized conductivity and hence heats more efficiently. For the t-test comparing DI water with the 841 nanorods, the two-tailed p-value is 0.0013, which is also lower than the p-value threshold for statistical significance. It can also be concluded that the normalized conductivity mean is different between DI water and 841 nanorods.

Samples	Number of trials	Average Normalized conductivity (degrees)	Standard deviation (degrees)	95% confidence interval
DI Water	3	8.832	0.163	0.184
762 nanorods 5mM	3	11.142	0.047	0.054
762 nanorods 2.5mM	3	10.630	0.107	0.122
762 nanorods 1.25mM	2	10.162	0.040	0.055
841 nanorods 5mM	3	9.983	0.184	0.208
841 nanorods 2.5mM	3	9.535	0.071	0.081
841 nanorods 1.25mM	3	9.514	0.106	0.121

Table 1a: Summary of data comparing the normalized conductivity of DI water, 762 nanorods and 841 nanorods at 5mM, 2.5 mM and 1.25 mM

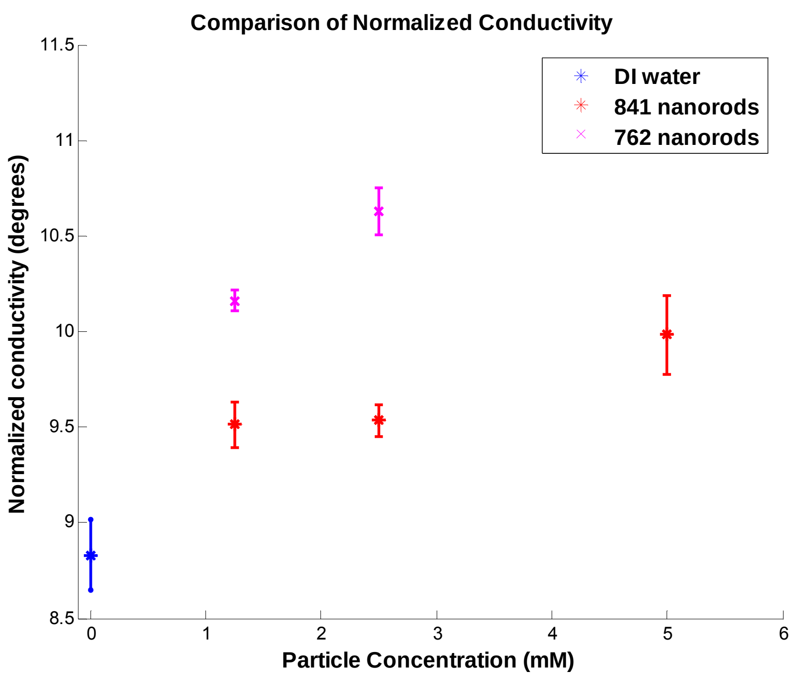


Figure 1: Average normalized conductivity vs. particle concentration with the 95% confidence interval

	Normalized conductivity (degrees)				
Samples	**Trial 1**	**Trial 2**	**Trial 3**	**Mean of the trials**	**Standard deviation**
DI Water	8.665	8.840	8.991	8.832	0.163
762 nanorods 5mM	11.195	11.105	11.127	11.142	0.047
				t	23.6
				p-value	<0.0001
				degrees of freedom	4

Table 1b: Two sample t-test is performed comparing the normalized conductivity obtained from DI water to that obtained from 5 mM concentration of 762 nanorods. The p-value shows that the two means indeed differ.

	Normalized conductivity (degrees)				
Samples	**Trial 1**	**Trial 2**	**Trial 3**	**Mean of the trials**	**Standard deviation**
DI Water	8.665	8.840	8.9908	8.832	0.163
841 nanorods 5mM	10.068	10.108	9.772	9.983	0.184
				t	8.1130
				p-value	0.0013
				degrees of freedom	4

Table 1c: Two sample t-test is performed comparing the normalized conductivity obtained from DI water to that obtained from 5 mM concentration of 841 nanorods. The p-value shows that the two means indeed differ.

Part II Data taken when UW staff was at NU

This set of data includes DI water, CTAB (surfactant used for making the nanorods) and five concentrations (5mM, 2.5 mM, and 1.25 mM, 0.625 mM and 0.3125 mM) of 762 nanorods and 841 nanorods. The CTAB used in the testing is at 50 mM concentration because it is estimated that the CTAB on the surface of the nanorods upon the completion of the synthesis is around 50 mM. In general, 1 trial is performed for each sample. The average normalized conductivity of each sample is listed in Table 2 and 3. Figure 2 shows a plot of the normalized conductivity vs. concentration.

The general trend is that the conductivity of the nanorods is higher than that of water. In addition, there seems to be a trend that as the concentration of the nanorods increases, the conductivity increases. In general, the experiment is pretty sensitive, so it is better to take an average of several experiment trials when determining the normalized conductivity. However, due to the time constraint of the UW staff visit, there is only one trial performed for each sample. The data taken before the UW staff came is more reliable because multiple trials were performed for each sample to obtain an average.

It is observed that CTAB has a higher conductivity than DI water and the nanorods samples. The 50 mM CTAB solution sample was made by dissolving CTAB in DI water and heating the mixture, and the mixture had to be completely cooled down before the heating experiment. When the CTAB sample was tested, some of the CTAB compound could be seen suspended in the solution. The higher conductivity could have been achieved because the CTAB was not completely dissolved in the solution, and “big clumps” of the CTAB particles were heated more efficiently. Also, the concentration of the CTAB solution is only an estimate.

Samples	**Number of trials**	**Average Normalized conductivity (degrees)**
DI water	2	10.427
CTAB (50 mM)	1	12.531
5 mM 841nanorods	2	11.727
2.5 mM 841 nanorods	1	10.931
1.25 mM 841 nanorods	1	11.222
0.625 mM 841 nanorods	1	11.011
0.3125 mM 841 nanorods	1	11.154

Table 2: Summary of data comparing the normalized conductivity of DI water, CTAB and 841 nanorods at 5mM, 2.5 mM and 1.25 mM, 0.625 mM and 0.3125 mM

Samples	Number of trials	Average Normalized conductivity (degrees)
DI water	2	10.427
CTAB (50 mM)	1	12.531
5 mM 762nanorods	1	11.917
2.5 mM 762 nanorods	1	12.192
1.25 mM 762 nanorods	1	11.651
0.625 mM 762 nanorods	2	12.019
0.3125 mM 762 nanorods	1	11.242

Table 3: Summary of data comparing the normalized conductivity of DI water, CTAB and 762 nanorods at 5mM, 2.5 mM and 1.25 mM, 0.625 mM and 0.3125 mM

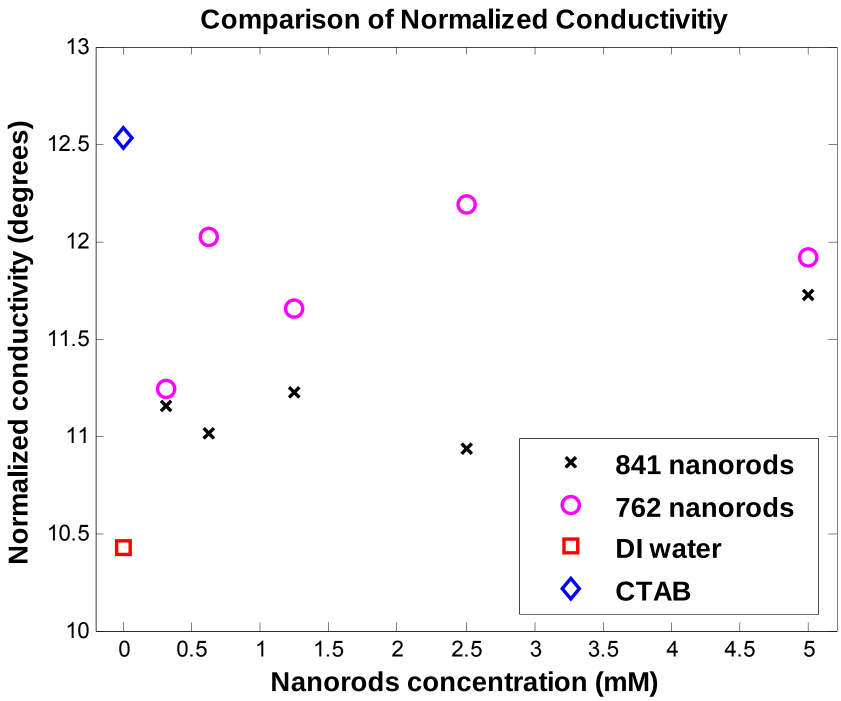


Figure 2: Normalized conductivity vs. particle concentration

Part 3: Conclusion

A heating effect is observed in both of the nanoparticles samples (762 nanorods and 841 nanorods) compared to DI water, and it seems that the effect is related to the concentration of the nanoparticles. Both types of nanoparticles at the highest available concentration (5 mM) have a higher normalized conductivity compared to water as shown by the two sample t-tests.

Experiments suggesting that gold nanoparticles at these concentrations may not be effective absorbers and that CTAB may be responsible for observed absorption.

This part of the report summarizes two results:

1) The results from microwave heating experiment comparing the heating effect of gold nanorods with several background constituent solutions, and

2) The result from a numerical study of equivalent permittivity of of ensembles of dielectric particles at different volume fractions.

This component of the report summarizes the result from the microwave heating experiment comparing the heating effect of gold nanorods with several background constituent solutions.

Microwave heating experiment comparing gold nanoparticles with background constituent solutions

The motivation behind the study is to examine whether the heating effect observed previously from the gold nanorod solutions is due to the gold nanorods or from the other background constituents in the solution. Four samples are tested in the heating experiment: a 5 mM concentration of gold nanorods with a plasmon peak at 870 nm, DI water, a 50 mM concentration of the surfactant CTAB which is used stabilize the particles in the solution and a supernatant solution from the gold nanorods solution after centrifuging. The concentration of CTAB is chosen to reflect the concentration of CTAB that surrounds the gold nanorods. The supernatant may contain various salts used in the synthesis of the nanorods. Figure 1 illustrates the temperature trace curves obtained from the experiment, and table 1 summarizes the average conductivity obtained for each solution.

The result from this study is that the background constituent liquids seem to heat just as efficiently as the gold nanorods. It is possible that the heating effect from the gold nanorods is not observed because the volume fraction of the gold nanorods is very low in the solution, and perhaps the gold nanorods cannot efficiently heat up the volume of solution as large as the one they are suspended in. One alternative is to test for a more localized heating effect from the gold nanorods rather than a global heating effect.

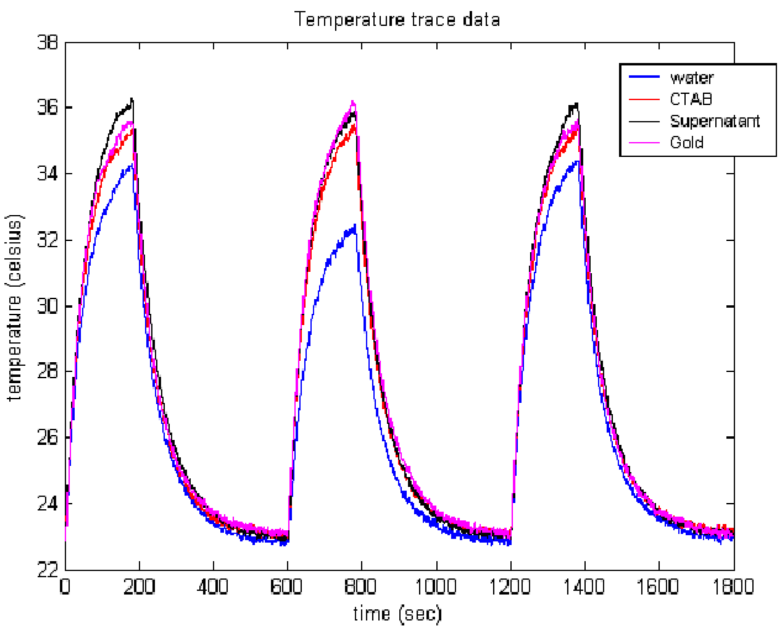


Figure 1: Comparison of temperature data from DI water, 50 mM CTAB, supernatant and 5 mM concentration of gold nanorods

Sample	Trial 1	Trial 2	Trial 3	Average conductivity	Std/average conductivity
DI water	11.055	13.340		12.198	0.132
CTAB	12.637	12.379		12.508	0.0146
Supernatant	13.467	16.862		15.164	0.158
Gold nanoparticles	12.912	14.179	13.919	13.670	0.0489

Table 1: Normalized conductivity obtained for each sample solution at different trials. The normalized conductivity is the temperature difference between the room temperature and the temperature the particles heat to during the heating cycle

Part 2: Calculation of the equivalent permittivity of dielectric spheres at different volume fractions

The equivalent permittivities for different ensembles of uniform dielectric spheres at different volume fractions are calculated using the Generalized Multiparticle Mie method. The dielectric spheres are illuminated by a x-polarized plane wave. Particle ensembles with 1-25% volume fraction are simulated, and multiple realizations for each volume fraction are generated in order to obtain the average property for the configuration. Since the particles are much smaller compared to the wavelength, the particles are treated as dipoles, and their internal field is used to approximate the polarization of the ensemble and used to calculate the equivalent permittivity. The particles simulated are 0.5 micron in radius, illuminated by a 2GHz microwave, and they have a refractive index of 1.59. Figure 2 shows the equivalent permittivities obtained for the different volume fraction ensembles calculated using the method described above. The result is also compared to the case when the internal field of a single particle is used to calculate the expected polarizations of different volume fraction ensembles, and the equivalent permittivities are calculated based on the internal field of the single particle. This allows for the comparison of the effect of particle interactions on the dielectric property at different volume fraction. The result is also compared to the dielectric properties calculated with the Maxwell Garnett mixing formula. The result of the comparisons shows that the contribution of particle interaction to the equivalent permittivity is small up to a 25% volume fraction.

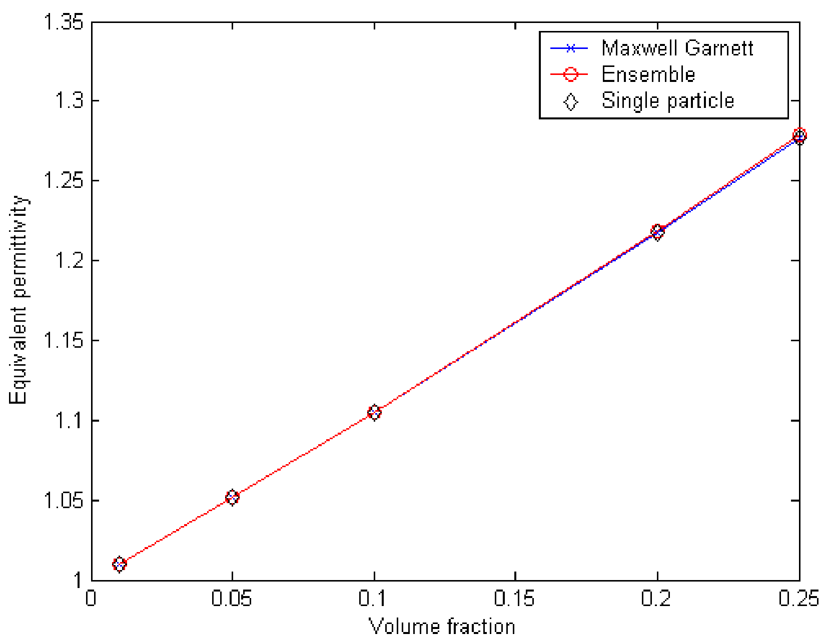


Figure 2: Equivalent permittivity of ensembles of dielectric spheres at 1-25% volume fractions. Comparison is made between the dielectric properties calculated with/without the inclusion of particle interaction and the Maxwell Garnett rule.

II) Summary of swept measurements using coaxial probe and cavity (UW apparatus)

Coaxial Probe and Cavity Measurements

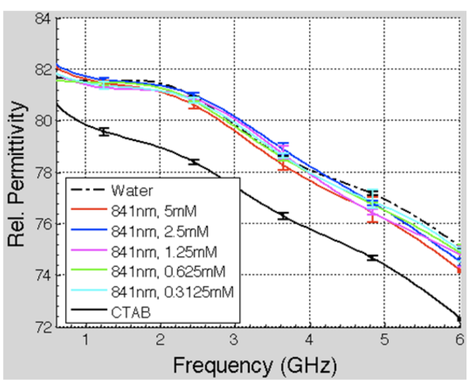
Nanoparticle Solutions

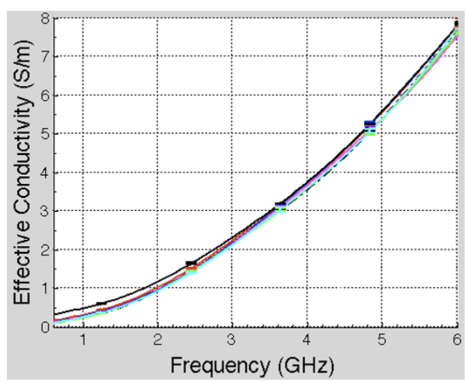
- Two gold nanoparticle solutions defined by their absorption spectrum
  - 841nm and 762nm
- Dispersion are composed of gold nanorods
  - ~4 to 1 aspect ratio
- Dispersed in CTAB-water solution
  - Concentration of CTAB solution ~ 50 mM (exact concentration not known)

Measurement summary

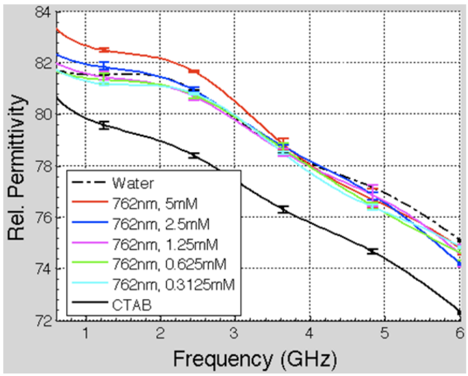
- Five different concentrations for each solution
  - Highest concentration: 5 mM
  - Solutions diluted with water
- Dielectric measurements made using both coaxial probe and cavity techniques
  - Coax probe before heating (0.5 GHz – 20 GHz)
  - Cavity before heating (2.19 GHz and 5 GHz)
  - Cavity after heating (2.19 GHz and 5 GHz)

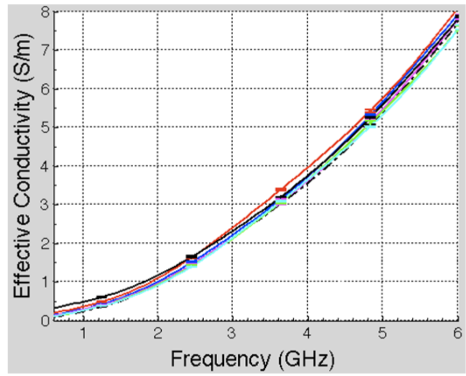
Dielectric Spectroscopy Results for 841nm nanoparticle solutions



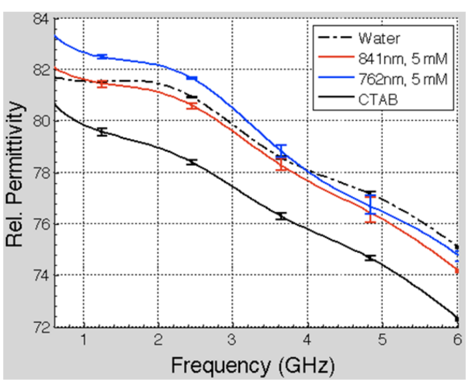


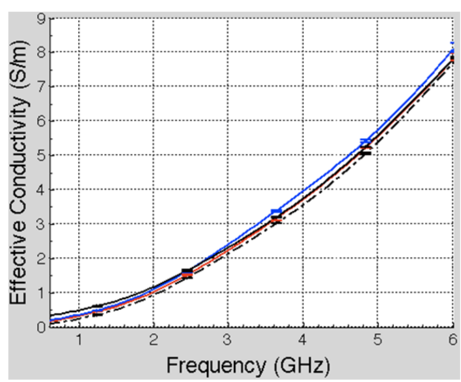
Dielectric Spectroscopy Results for 762nm nanoparticle solutions





Comparison of 841nm and 762nm solutions





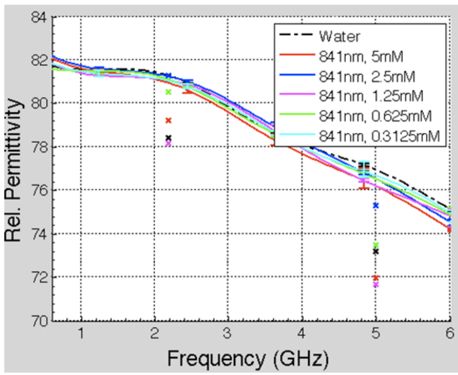
Dielectric Spectroscopy Data Summary at 4 GHz

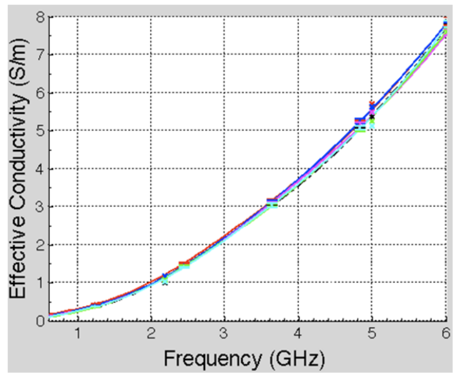
Sample	Average ε_r (4 GHz)	Average σ_{eff} (4 GHz)
Water	78.1	3.54
CTAB	75.8	3.73
841nm, 5 mM	77.7	3.71
841nm, 2.5 mM	78.3	3.71
841nm, 1.25 mM	78.0	3.66
841nm, 0.625 mM	78.0	3.7
841nm, 0.3125 mM	78.0	3.6
762nm, 5 mM	78.1	3.94
762nm, 2.5 mM	78.2	3.71
762nm, 1.25 mM	78.0	3.60
762nm, 0.625 mM	78.0	3.63
762nm, 0.3125 mM	77.8	3.63

Cavity measurement summary

Sample	ε_r (2.19 GHz)	σ_{eff} (2.19 GHz)	ε_r (5 GHz)	σ_{eff} (5 GHz)
Water	78.4	1.00	73.19	5.36
841nm, 5 mM	79.2	1.18	72.0	5.71
841nm, 2.5 mM	81.3	1.16	75.3	5.62
841nm, 1.25 mM	78.1	1.06	71.7	5.49
841nm, 0.625 mM	76.6	1.06	70.8	5.24
841nm, 0.3125 mM	80.5	1.01	73.5	5.11
762nm, 5 mM	78.9	1.21	72.6	5.88
762nm, 2.5 mM	78.1	1.11	73.5	5.37
762nm, 1.25 mM	78.1	1.06	73.5	5.49
762nm, 0.625 mM	81.3	1.06	74.4	5.75
762nm, 0.3125 mM	80.5	1.06	74.4	5.88

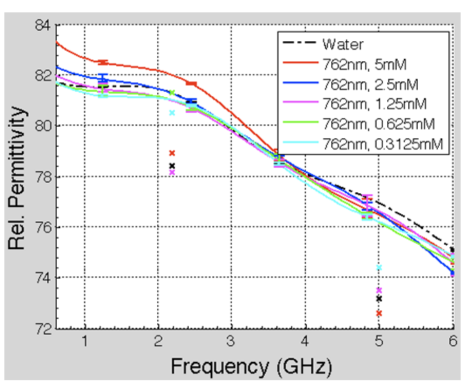
Cavity measurement data for 841nm samples

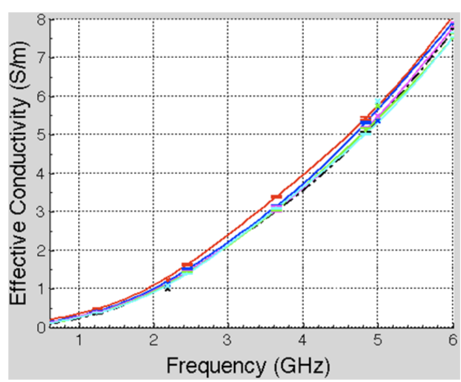




- x – cavity data points

Cavity measurement data for 762nm samples





- x – cavity data points

Cavity measurement summary for samples after heating – 841nm

Sample	ε_r (2.19 GHz)	σ_{eff} (2.19 GHz)	ε_r (5 GHz)	σ_{eff} (5 GHz)
841nm, 5 mM Before heating	79.2	1.18	72.0	5.71
841nm, 5 mM After heating	78.9	1.16	68.96	5.75
841nm, 2.5 mM Before heating	81.3	1.16	75.3	5.62
841nm, 2.5 mM After Heating	78.93	1.11	74.40	5.49
841nm, 1.25 mM Before heating	78.1	1.06	71.7	5.49
841nm, 1.25 mM After Heating	78.15	1.06	73.49	5.75
841nm, 0.625 mM Before heating	76.6	1.06	70.8	5.24
841nm, 0.625 mM After Heating	79.72	1.06	75.30	5.88
841nm, 0.3125 mM Before heating	80.5	1.01	73.5	5.11
841nm, 0.3125 mM After heating	78.93	0.97	72.59	5.11

Cavity measurement summary for samples after heating – 762nm

Sample	ε_r (2.19 GHz)	σ_{eff} (2.19 GHz)	ε_r (5 GHz)	σ_{eff} (5 GHz)
762nm, 5 mM Before heating	78.9	1.21	72.6	5.88
762nm, 5 mM After heating	79.7	1.16	72.6	5.88
762nm, 2.5 mM Before heating	78.1	1.11	73.5	5.37
762nm, 2.5 mM After heating	78.9	1.11	72.6	5.75
762nm, 1.25 mM Before heating	78.1	1.06	73.5	5.49
762nm, 1.25 mM After heating	78.2	1.06	76.2	5.75
762nm, 0.625 mM Before heating	81.3	1.06	74.4	5.75
762nm, 0.625 mM After heating	80.5	1.11	73.5	5.75
762nm, 0.3125 mM Before heating	80.5	1.06	74.4	5.88
762nm, 0.3125 mM After heating	80.5	1.06	74.4	5.37

Conclusions

- No significant changes in dielectric properties between different solutions.
- Cavity measurements show that there are no significant changes in dielectric properties after samples were heated.